Determination of P, Ca, Mg, K, Na, NH_4

Short Test Methods Used in Soil Testing Division,
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DETERMINATION OF P, Ca, Mg, K, Na, NH_4

Extraction

Extracting solution: Approximately 0.05N HCl and 0.025N H_2SO_4 mixture. Measure about 15 liters of H_2O into a 20-liter Pyrex bottle. Add 14.0 ml conc. H_2SO_4 and 83 ml conc. HCl, make up to 20 liters with H_2O and mix.

Procedure: Measure 1 scoop (4-ml capacity corresponding to about 5 g) of soil passed through a 2-mm sieve into a 50-ml Erlenmeyer flask. Add 1 scoop (about 250 mg) of charcoal and then 20 ml of extracting solution. Shake in a mechanical shaker for 5 minutes and filter. In the extract, determine P, Ca, Mg, K, Na and NH_4.

Calcium and Phosphorus

Reagents:

1. Oxalic acid – Triethanolamine mixture. Dissolve 6 g oxalic acid in about 700 ml of H_2O, add 50 ml of commercial triethanolamine and make up to one liter with H_2O. [During summer months store in refrigerator and use while cold.]

2. Ammonium molybdate – Ammonium vanadate mixture. Dissolve 25 g ammonium molybdate in 500 ml of H_2O. Dissolve 1.25 g ammonium vanadate in 500 ml of 1:1 HNO_3 and mix equal volumes of these solutions. Prepare mixture fresh each week.

3. Calcium standard – 0.02N Ca. Dissolve 1.009 g $CaCO_3$ (Iceland spar) in one liter of extracting solution.

4. Phosphorus standard – 25 ppm P. Dissolve 0.1098 g KH_2PO_4 in one liter of extracting solution.

Ca and P standardization

Reagent 3 ml Ca	Reagent 4 ml P	Cenco Photelometer readings		Ca in Soil meq /100g	Ca in Soil CaO lb/acre	P in Soil ppm P	P in Soil P_2O_5 lb/acre
0.0	4.0	100	16	0.0	0	100.0	456
0.5	3.5	64	19	1.0	560	87.5	400
1.0	3.0	33	23	2.0	1120	75.0	342
2.0	2.0	13	31	4.0	2240	50.0	228
3.0	1.0	9	51	6.0	3360	25.0	114
3.5	0.5	8	68	7.0	3920	12.5	56
4.0	0.0	6	100	8.0	4480	0.0	0
Add Reagent 1 and continue as given under instrument setting and procedure.							

Instrument setting of the Cenco Photelometer: Adjust to zero by means of the adjuster. Switch to "on" position. Allow to warm up for 15 minutes. Insert vial containing 4 ml extracting solution, 1 ml each of reagents 1 and 2. Adjust to 100 by means of the metal knob. Use a purple filter (420 μ).

Procedure: Measure into glass vials* 4 ml of the soil extract, add 1 ml of Reagent 1 and mix. After 10 minutes and no longer than 2 hours, mix (by inverting the vial) and measure the transmission of light and the turbid solution. (The turbidity developed is a measure of Ca.) See Table 1 for conversion of readings to meq Ca per 100 g soil.

Add 1 ml of Reagent 2 and read after 20 minutes or longer. (The color developed is a test for P.) See Table 2 for conversion of readings to ppm P in the soil.

If in the test for Ca, the photelometer reading is less than 10, repeat by using a smaller aliquot diluted with extracting solution to 4 ml. Proceed as above and multiply the results by the appropriate dilution factor.

Magnesium

Stock solutions:

1. CaCl2, 2%. Dissolve 20 g $CaCl_2 \cdot 2H_2O$ in one liter of H_2O.

2. Hydroxylamine hydrochloride, 5%. Dissolve 50 g of the salt in one liter of H_2O.

3. Aluminum chloride, 1%. Dissolve 10 g $AlCl_3 \cdot 6H_2O$ in one liter of H_2O.

4. Thiazol yellow, 0.3%. Dissolve 3.0 g Thiazol yellow in one liter of H_2O.

5. Sodium hydroxide, 30%. Dissolve 300 g NaOH in one liter of H_2O.

Reagent A: Measure about 800 ml H_2O into a liter flask and add in the following order: 20 ml each of stock solutions 1, 2 and 3; and 50 ml of triethanolamine. Make up to volume with H_2O and mix.

Reagent B: Measure about 900 ml H_2O into a liter flask. Add 20 ml of stock solution 4 and 50 ml of stock solution 5. Make up to volume and mix.

Mg solutions for standardization: Dissolve 1.2325 g $MgSO_4 \cdot 7H_2O$ in one liter of extracting solution. This is 0.01N. Prepare the range of standards as follows (use 50-ml volumetric flasks).

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* Capacity of these vials is 8 ml, length 60 mm, and diameter 17 mm.

Mg solution (ml)	Extracting solution (ml)	Cenco Photelometer readings	Mg in soil	
			meq/100 g	MgO (lb/acre)
0.0	50.0	100	0.0	0
2.5	47.5	77	0.2	80
5.0	45.0	63	0.4	160
10.0	40.0	48	0.8	320
15.0	35.0	41	1.2	480
20.0	30.0	39	1.6	640

Procedure: Measure 1-ml portions of the Mg standard solutions or the soil extract into vials, add 2 ml of Reagent A, mix and then add 2 ml of Reagent B. Mix and take photelometer readings within 5 minutes. Use a green filter. For the 100 setting, use 1 ml of extracting solution containing 2 ml each of Reagents A and B. See Table 3 for conversion of readings to meq Mg per 100 g soil. Use a smaller aliquot if the readings are less than 40. In this case, add sufficient extracting solution to a volume of 1 ml. Proceed as above and multiply the results by the appropriate dilution factor.

Potassium — Flame Photometric (P. E. 52-A)

K — standard solutions and calibration curve

KCl - 0.005N. Dissolve 0.3728 g of dried KCl in one liter of extracting solution.

Use the following amounts of KCl and make up to 100 ml with extracting solution (ml)	Flame Photometer readings (direct)	K in soil	
		meq/100 g	K_2O (lb/acre)
0.0	0	0.0	0
2.5	9	0.05	47
5.0	27	0.10	94
7.5	44	0.15	141
10.0	65	0.20	188
12.5	84	0.25	235
15.0	100	0.30	282

Procedure: Put the instrument into operation based on the instructions of the manual supplied by the manufacturer. Run a standard curve on the above solutions. When measuring the unknown, check performance with standard solution after every four to six samples. To do this, use the standard nearest the last measured unknown. If the

concentration of K is too high, dilute an aliquot with extracting solution. See Table 4 for conversion of readings to meq K per 100 g soil.

Sodium — Flame Photometric

Na standard solutions and calibration curve

NaCl - 0.005N. Dissolve 0.2923 g of dried NaCl in one liter of extracting solution.

Use the following amounts of NaCl and make up to 100 ml with extracting solution (ml)	Flame Photometer readings (direct)	Na in soil	
		meq/100 g	Na_2O (lb/acre)
0.0	0	0.0	0
2.5	16	0.05	31
5.0	35	0.10	62
7.5	54	0.15	93
10.0	71	0.20	124
12.5	87	0.25	155
15.0	100	0.30	186

See Table 5 for conversion of readings on soil extract to meq Na per 100 g soil.

Procedure: Select the appropriate wave length for the Na line and proceed as in the determination of K.

Ammonia Nitrogen

Reagents:

1. Sodium hydroxide-tartrate solution. Dissolve 40 g sodium tartrate in about 600 ml of H_2O, add 13 g NaOH. Dissolve and make up to one liter with H_2O.

2. Nessler reagent. Measure into a 500-ml volumetric flask, 5 g mercuric iodide and 3.5 g potassium iodide in as little water as needed. Add 11 g KOH, dissolve and make up to volume with H_2O. Store in brown bottle. Dissolve in a 500-ml volumetric flask, 5 g powdered gum acacia by adding just sufficient H_2O to make a paste. Dilute with H_2O to volume. Store in brown bottle. After a few days, remove 25 ml of each solution and dilute to 400 ml with H_2O. Store in a brown bottle.

3. Standard ammonium nitrogen solution (25 ppm N and standard curve). Dissolve 0.0588 g $(NH_4)_2SO_4$ in extracting solution made up to 500 ml.

Add the following amounts of $(NH_4)_2SO_4$ solution to 50-ml volumetric flasks and make up to volume with extracting solution.

$(NH_4)_2SO_4$ solution (ml)	Cenco Photelometer readings	N in soil	
		ppm	lb/acre
0	100	0	0
10	64	20	40
20	46	40	80
30	36	60	120
40	30	80	160

Procedure: Measure 1 ml of the soil extract into 8-ml capacity vials, add 2 ml reagent 1 and 2 ml reagent 2. Mix and read, after 15 minutes, the transmittance of the photelometer using the purple filter (420 μ). Take a smaller aliquot and dilute with extracting solution if the photelometer readings are less than 40. See Table 6 for conversion of readings to ppm NH_4-N.

Organic Matter

Reagents:

1. Dichromate solution – 0.4N. Dissolve 360 g sodium dichromate and make up to 18 liters with H_2O.

2. Ferrous ammonium sulfate – 0.4N. Add to about 12 liters of H_2O, 360 ml conc. H_2SO_4, dissolve 2880 g ferrous ammonium sulfate. Make up to 18 liters with H_2O and mix thoroughly.

Procedure: Measure one scoop (approximately 1.5 g) of soil into a 250 ml Erlenmeyer flask, and add — by means of an automatic delivery pipette — 25 ml sodium dichromate solution and 25 ml H_2SO_4 (tech), under a hood. Allow to stand. Add approximately 100 ml H_2O and allow to cool. Add five drops Ortho-Penanthroline indicator, titrate with ferrous ammonium sulfate solution. From a "blank" run of solutions, calculate the %O.M. by subtracting the titer of the unknown from the "blank" and multiply by 0.2.

pH

Procedure: Measure approximately 10 g soil (8-cm^3 scoop) into a 50-cm^3 beaker, and add 10 cm^3 distilled water. Read on glass electrode assembly after one hour. Stir while reading or immediately before.

Table 1. Conversion of Cenco Photelometer readings into meq Ca per 100 g soil					
	0	**2**	**4**	**6**	**8**
0		8.5	8.0	7.6	7.4
1	7.2	7.0	6.8	6.6	6.4
2	6.2	6.0	5.8	5.6	5.4
3	5.2	5.0	4.8	4.6	4.4
4	4.2	4.0	3.8	3.6	3.4
5	3.2	3.0	2.8	2.6	2.5
6	2.3	2.15	2.0	1.8	1.7
7	1.55	1.4	1.3	1.1	1.0
8	0.9	0.8	0.7	0.55	0.5
9	0.4	0.3	0.2	0.1	0.05

Table 2. Conversion of Cenco Photelometer readings into ppm P (soil basis)					
	0	**2**	**4**	**6**	**8**
1	140	122	110	100	92
2	84	78	70	64	55
3	53	49	46	43	40
4	38	36	33	31	28
5	26	24	23	21	19
6	18	17	15	14	13
7	12	11	10	9	8
8	7	6	5	4	3.5
9	3	2.5	2	1.5	1

Table 3. Conversion of Cenco Photelometer readings into meq Mg per 100 g soil					
	0	**2**	**4**	**6**	**8**
3	2.20	1.82	1.60	1.40	1.28
4	1.20	1.10	1.02	0.94	0.82
5	0.80	0.72	0.66	0.60	0.54
6	0.50	0.48	0.44	0.40	0.34
7	0.32	0.28	0.26	0.22	0.20
8	0.18	0.16	0.14	0.12	0.10
9	0.08	0.06	0.04	0.03	0.02

Table 4. Conversion of flame photometer readings into meq K per 100 g soil					
	0	**2**	**4**	**6**	**8**
0	0.000	0.020	0.030	0.040	0.050
1	0.055	0.060	0.065	0.070	0.075
2	0.080	0.085	0.090	0.095	0.100
3	0.110	0.115	0.120	0.125	0.130
4	0.140	0.145	0.150	0.155	0.160
5	0.165	0.170	0.175	0.180	0.185
6	0.190	0.195	0.200	0.205	0.210
7	0.215	0.220	0.225	0.230	0.235
8	0.240	0.245	0.250	0.255	0.260
9	0.270	0.275	0.280	0.285	0.290
10	0.300				

Table 5. Conversion of flame photometer readings into meq Na per 100 g soil					
	0	**2**	**4**	**6**	**8**
0	0.000	0.010	0.015	0.020	0.025
1	0.030	0.035	0.040	0.045	0.050
2	0.060	0.065	0.070	0.075	0.080
3	0.085	0.090	0.095	0.100	0.105
4	0.110	0.115	0.120	0.125	0.130
5	0.135	0.140	0.150	0.155	0.160
6	0.165	0.170	0.175	0.180	0.190
7	0.195	0.200	0.205	0.210	0.220
8	0.225	0.230	0.240	0.245	0.250
9	0.260	0.265	0.270	0.280	0.290
10	0.300				

Table 6. Conversion of Cenco Photelometer readings into ppm NH_4–N (soil basis)					
	0	**2**	**4**	**6**	**8**
3	80	70	65	60	55
4	50	47	43	40	36
5	33	30	28	26	24
6	22	20	19	18	16
7	15	14	13	12	11
8	10	9	8	7	6
9	5	4	3	2	1